

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
 United States Patent and Trademark
 Office
 Box PCT
 Washington, D.C.20231
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 18 October 2000 (18.10.00)	
International application No. PCT/GB00/00524	Applicant's or agent's file reference CTV/P45103WO
International filing date (day/month/year) 17 February 2000 (17.02.00)	Priority date (day/month/year) 17 February 1999 (17.02.99)
Applicant RAMSHAW, Colin et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
 15 September 2000 (15.09.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Zakaria EL KHODARY Telephone No.: (41-22) 338.83.38
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PATENT COOPERATION TREATY

PCT

NOTIFICATION OF THE RECORDING
OF A CHANGE(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
ROYAUME-UNI

Date of mailing (day/month/year) 27 November 2000 (27.11.00)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference CTV/P45103WO	
International application No. PCT/GB00/00524	International filing date (day/month/year) 17 February 2000 (17.02.00)

1. The following indications appeared on record concerning:		
<input checked="" type="checkbox"/> the applicant	<input type="checkbox"/> the inventor	<input type="checkbox"/> the agent
<input type="checkbox"/> the common representative		
Name and Address UNIVERSITY OF NEWCASTLE 6 Kensington Terrace Newcastle upon Tyne NE1 7RU United Kingdom	State of Nationality GB	State of Residence GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	
2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:		
<input checked="" type="checkbox"/> the person	<input checked="" type="checkbox"/> the name	<input checked="" type="checkbox"/> the address
<input type="checkbox"/> the nationality		
<input type="checkbox"/> the residence		
Name and Address NEWCASTLE UNIVERSITY VENTURES LIMITED Sun Alliance House 35 Mosley Street Newcastle upon Tyne NE1 1XX United Kingdom	State of Nationality GB	State of Residence GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	
3. Further observations, if necessary:		
4. A copy of this notification has been sent to:		
<input checked="" type="checkbox"/> the receiving Office	<input type="checkbox"/> the designated Offices concerned	
<input type="checkbox"/> the International Searching Authority	<input checked="" type="checkbox"/> the elected Offices concerned	
<input checked="" type="checkbox"/> the International Preliminary Examining Authority	<input type="checkbox"/> other:	

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Maria Victoria CORTIELLO
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

NOTIFICATION OF THE RECORDING
OF A CHANGE(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
ROYAUME-UNI

Date of mailing (day/month/year) 22 August 2001 (22.08.01)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference CTV/P45103WO	
International application No. PCT/GB00/00524	International filing date (day/month/year) 17 February 2000 (17.02.00)

1. The following indications appeared on record concerning:

☒ the applicant ☐ the inventor ☐ the agent ☐ the common representative

Name and Address NEWCASTLE UNIVERSITY VENTURES LIMITED Sun Alliance House 35 Mosley Street Newcastle upon Tyne NE1 1XX United Kingdom	State of Nationality GB	State of Residence GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

☒ the person ☐ the name ☐ the address ☐ the nationality ☐ the residence

Name and Address PROTENSIVE LIMITED 35 Hills Road Cambridge CB2 1NT United Kingdom	State of Nationality GB	State of Residence GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

<input checked="" type="checkbox"/> the receiving Office	<input type="checkbox"/> the designated Offices concerned
<input type="checkbox"/> the International Searching Authority	<input checked="" type="checkbox"/> the elected Offices concerned
<input checked="" type="checkbox"/> the International Preliminary Examining Authority	<input type="checkbox"/> other:

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer CORTIELLO Maria Victoria Telephone No.: (41-22) 338.83.38
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PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference CTV/P45103W0	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 00/ 00524	International filing date (day/month/year) 17/02/2000	(Earliest) Priority Date (day/month/year) 17/02/1999
Applicant UNIVERSITY OF NEWCASTLE et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of Invention is lacking** (see Box II).

4. With regard to the **title**,

☐ the text is approved as submitted by the applicant.

☒ the text has been established by this Authority to read as follows:

PROCESS FOR THE CONVERSION OF A FLUID PHASE SUBSTRATE BY DYNAMIC HETEROGENEOUS CONTACT WITH A SOLID AGENT

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawing** to be published with the abstract is Figure No.

☒ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

3

☐ None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/00524

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01J19/18 B01J19/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 499 362 A (TIOXIDE GROUP SERVICES LTD) 19 August 1992 (1992-08-19) cited in the application the whole document ---	1,2,4,7, 9,10
X	WO 96 00189 A (CHRISTENSEN PAUL A, HUTSON GRAHAM V) 4 January 1996 (1996-01-04) page 11, line 3 -page 14, line 5 page 19, line 25 -page 21, line 7 claims 1,13-15,21,22,27-33; figures 3,4 ---	1,3,4,6, 7,9-11
A	US 4 356 133 A (COWEN GEOFFREY, NORTON-BERRY PHILIP, STEEL MARGARET L) 26 October 1982 (1982-10-26) column 2, line 1 - line 6 column 3, line 53 -column 4, line 13 column 4, line 53 -column 5, line 11 figure 1 --- -/--	1,4,9-12



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "I" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

22 June 2000

Date of mailing of the international search report

10/07/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
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Authorized officer

Vlassis, M

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/00524

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1 600 708 A (G.V.PLANER LIMITED) 21 October 1981 (1981-10-21) claims 1,5,7,16; figures 1,2 -----	2

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 00/00524

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0499362	A	19-08-1992	AU 646133 B	10-02-1994
			AU 1037992 A	13-08-1992
			CA 2059932 A	10-08-1992
			DE 69203877 D	14-09-1995
			DE 69203877 T	07-12-1995
			DK 499362 T	18-09-1995
			ES 2075602 T	01-10-1995
			GB 2252707 A, B	12-08-1992
			US 5308458 A	03-05-1994
WO 9600189	A	04-01-1996	AU 2680895 A	19-01-1996
			EP 0766647 A	09-04-1997
			JP 10502863 T	17-03-1998
			US 5932111 A	03-08-1999
US 4356133	A	26-10-1982	AU 523498 B	29-07-1982
			AU 4446779 A	30-08-1979
			CA 1125995 A	22-06-1982
			DE 2960714 D	26-11-1981
			EP 0003908 A	05-09-1979
			IT 1202891 B	15-02-1989
			JP 1437024 C	25-04-1988
			JP 54163784 A	26-12-1979
			JP 62043734 B	16-09-1987
GB 1600708	A	21-10-1981	NONE	

09/913966

(2)

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
GRANDE BRETAGNE

10.MAY.2001*054130

PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing
(day/month/year)

08.05.2001

Applicant's or agent's file reference
CTV/P45103WO

IMPORTANT NOTIFICATION

International application No.
PCT/GB00/00524

International filing date (day/month/year)
17/02/2000

Priority date (day/month/year)
17/02/1999

Applicant

NEWCASTLE UNIVERSITY VENTURES LTD. et al

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the International preliminary examination report and its annexes, if any, established on the International application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.



Name and mailing address of the IPEA/



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Authorized officer

Gregoire, J-P

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PATENT COOPERATION TREATY

PCT

09/913966

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference CTV/P45103WO		FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/GB00/00524	International filing date (day/month/year) 17/02/2000	Priority date (day/month/year) 17/02/1999	
International Patent Classification (IPC) or national classification and IPC B01J19/18			
Applicant NEWCASTLE UNIVERSITY VENTURES LTD. et al			



10.MAY2001*054731

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets, including this cover sheet.
- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 15/09/2000	Date of completion of this report 08.05.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tlx 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Buesing, G Telephone No. +49 89 2399 8356 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB00/00524

I. Basis of the report

1. With regard to the elements of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1,3,5-13,15	as originally filed		
2,4,14	as received on	23/03/2001 with letter of	21/03/2001

Claims, No.:

1-19	as received on	23/03/2001 with letter of	21/03/2001
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Drawings, sheets:

1/4-4/4	as originally filed
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2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**International application No. **PCT/GB00/00524**

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims 1 - 19
	No:	Claims
Inventive step (IS)	Yes:	Claims 1 - 19
	No:	Claims
Industrial applicability (IA)	Yes:	Claims 1 - 19
	No:	Claims

- 2. Citations and explanations**
see separate sheet

INTERNATIONAL PRELIMINARY

International application No. PCT/GB00/00524

EXAMINATION REPORT - SEPARATE SHEET

Section V:

1. The invention relates to the conversion of a fluid phase substrate in a spinning wheel reactor. An essential feature of the invention is the combination of the spinning wheel and the application of additional vibratory energy to the substrate.
2. None of the cited prior art documents teach or fairly suggest such a combination. As the claimed combination of features solves the problem to improve product quality, such as higher purity of a narrower molecular distribution in polymers, an inventive step is acknowledged.

EP 0 499 363 (Tioxide Group Services Limited) discloses another use for spinning disc technology in photo catalytic degradation of organic materials such a hydrocarbons. A solution of salicylic acid and titanium dioxide catalyst was passed
5 across the surface of a rotating disc and irradiated with ultra violet light.

These publications therefore disclose the use of spinning disc technology for heating and mass transfer in inert and reactive systems.

10 We have now surprisingly found that spinning disc technology may be further adapted to apply process intensification methods not only within the fields of heat and mass transfer but also within the field of heterogeneous contacting.

We have further surprisingly found that the quality of the product obtained is of
15 higher quality than that obtained by conventional processing having, for example, a higher purity or, in polymers, a narrower molecular distribution.

In addition to this, spinning disc technology can be used to obtain products not readily obtainable by other technology.

20 Accordingly in its broadest aspect there is provided according to the present invention a process for the conversion of a substantially fluid phase substrate by heterogeneous contact of the substrate or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a
25 surface of a support element or part thereof and the support element is adapted to rotate around an axis such that the solid phase agent provides a rotating surface or part thereof and the substrate provides a film flowing substantially radially outward from the axis in dynamic contact with the agent; characterised in that additional vibrational energy is applied to the substrate.

30 Reference herein to a rotating surface is to any continuous or discrete planar or three dimensional surface or assembly which rotates approximately or truly about an axis, and preferably is reference to an approximate or true rotating surface of revolution. An approximate rotating surface of revolution may comprise an asymmetric axis
35 and/or deviation in the surface body and/or circumference creating an axially or radially undulating surface of revolution. A discrete surface may be in the form of a mesh, grid, corrugated surface and the like.

value higher chain (C_{10+}) to high value lower chain branched (C_{2-6}) hydrocarbons, upgrading by decomposition (for example breakdown of salicylic acid) and the like. The process is also admirably suited for adaptation of existing homogeneous processes such as alkylation to form branched hydrocarbons and ethers, to
5 heterogeneous alternatives, using solid acids such as zeolites and the like.

Alternatively the process is a process for the crystallisation of a salt from a solution formed prior to or during contact with the rotating surface.

10 The process may therefore comprise a conversion of one substrate with itself in the presence of an agent as hereinbefore defined or may comprise the conversion of one substrate with one or more additional substrates in the presence of an agent as hereinbefore defined. Substrate(s) is fluid and may be gaseous, vaporous, liquid or fine flowable solid.

15 The conversion may be carried out entirely or in part under the influence of an added energy source which may be a source of thermal or radiation energy such as ultra violet, micro wave, infra-red, x-ray, or at elevated pressure or under the influence of electric or magnetic fields.

20 The process of the invention as hereinbefore defined may operated in a single or plural stages. A plural stage conversion may comprise a first pre-conversion stage with further post conversion or upgrading stages, and may be carried out batchwise with use of a single rotating surface as hereinbefore defined or may be carried out in
25 continuous manner with multiple rotating surfaces in series.

The process is particularly suited to continuous conversion, and may provide means to perform hitherto batch wise conversions in continuous manner, by means of the improved conversion efficiency and regulated contact time obtained with the
30 invention.

For example conversions requiring extended contact time may be carried out in continuous manner with use of a recycle of fluid exiting at the periphery of the rotating disc towards the axis of the rotating disc enabling sequential passes of fluid
35 across the surface. In continuous steady state operation an amount of fluid exiting the disc may be drawn off as product and an amount may be returned by recycle for further conversion with an amount of fresh substrate feed.

The spinning disc apparatus used is as described in Example 1. Calcium hydroxide and carbon dioxide (gas/liquid) were reacted on the surface of the disc and the effect of rotational speed on the crystal size and size distribution was studied.

- 5 Figure 1 illustrates a conventional spinning disc apparatus which is suited for adaptation according to the present invention. The apparatus is enclosed in vessel (1) having at its axis a drive shaft (2) supporting a spinning disc (3). Feed means (4) provides substrate to the surface of the disc (5) at its axis (6). Rotation of the disc causes substrate to flow radially outwards, whereby it contacts the surface of the
10 spinning disc. Fluid is collected at the peripheral edges of the disc by means of collection trough (7) and may be rapidly quenched by means of cooling coils (8). A skirt (9) prevents meniscal draw back of fluid contaminating the drive shaft mechanism. Inlet means (10) enable controlled environment conditions to be provided, for example a nitrogen atmosphere. Outlet vent means (11) enable the
15 venting of atmospheric gases or gases evolved during operation. Observation means are provided by means of windows (12) to observe the progress of the conversion.

- In Figure 2 is shown a typical spinning disc in the form of a rotating surface according to the present invention comprising an immobilised agent on its surface.
20 The disc (3) is illustrated without any additional surface features and comprises uniform coating (33) which is fused by chemical interaction (32) with the disc surface.

- In Figure 3 is shown an alternative spinning disc in which the agent which it is desired to immobilise is also known as a conventional support material, for example
25 a refractory oxide. In this case the spinning disc (3) is constructed entirely of refractory oxide, the body of which (34) provides a support function and the surface of which (35) provides a catalytic function.

- 30 In the process of the invention an apparatus of Figure 1 is started up and operated as described in Example 1. In the case that the process is an exothermic conversion, cooling coils (8) may be used to quench the collected product in the trough (7). The spinning disc is provided with heating coils (not shown) which may be used to initiate or maintain conversion. The disc or the reactor vessel may be provided with
35 a source of radiation, means for applying an electric or magnetic field and the like as described, at or above the disc surface or at the wall of the reactor vessel.

CLAIMS:

1. A process for the conversion of a substantially fluid phase substrate (15) by heterogeneous contact of the substrate (15) or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a surface (5,33,35) of a support element (3) or part thereof and the support element (3) is adapted to rotate around an axis (6) such that the solid phase agent provides a rotating surface (5,33,35) or part thereof and the substrate (15) provides a film (17) flowing substantially radially outward from the axis (6) in dynamic contact with the agent; characterised in that additional vibrational energy is applied to the substrate (15).
2. A process according to claim 1, wherein the additional vibrational energy is applied to the substrate (15) when on the rotating surface (5,33,35).
3. A process according to claim 1 or 2, wherein the additional vibrational energy is applied to the substrate (15) as it is being supplied to the rotating surface (5,33,35).
4. A process according to claim 1, 2 or 3, wherein the additional vibrational energy is applied to the substrate (15) after it has flowed across the rotating surface (5,33,35).
5. A process according to any preceding claim, wherein the additional vibrational energy is applied as ultrasound.
6. A process according to any preceding claim, wherein the rotating surface (5,33,35) is mechanically vibrated.
7. A process according to claim 6, wherein the rotating surface (5,33,35) is mounted off-centre on the axis of rotation (6).
8. A process according to claim 6 or 7, wherein the surface (5,33,35) is flexibly mounted on the support element (3).
9. A process according to claim 6, 7 or 8, wherein a mechanical vibrator is attached to the surface (5,33,35) or the support element (3).

10. A process according to claim 5, wherein the axis (6) is substantially vertical with the support element (3) adapted to rotate thereabout with the surface (5,33,35) uppermost, and wherein ultrasound is applied to the substrate (15) from an ultrasonic emitter located above the surface (5,33,35).
- 5
11. A process according to any previous claim, wherein the solid phase agent is in the form of a mesh, grid or corrugated surface.
12. A process according to any previous claim, wherein the solid phase agent comprises a nucleation or growth agent adapted for fluid phase substrate conversion by phase change to form crystals or grow seed crystals.
- 10
13. A process according to any one of claims 1 to 11, wherein the solid phase agent comprises a reagent, catalyst or initiator adapted for fluid phase substrate conversion by reaction to form products.
- 15
14. A process according to claim 13, wherein the solid phase agent is a zeolite.
15. A process according to claim 13, wherein the solid phase agent is a refractory oxide (34,35).
- 20
16. A process according to claim 13, wherein the solid phase agent is a sol gel.
17. A process according to claim 13, wherein the solid phase agent is Phillips catalyst.
- 25
18. A process according to any one of claims 12 to 17, wherein the solid phase agent is sprayed onto the surface (5,33,35).
- 30
19. A process according to any preceding claim, wherein additional thermal or radiation energy, including UV, IR, microwave, RF, X-ray, electric fields and magnetic fields, is applied to the substrate (15).



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)


(51) International Patent Classification: B01J 19/18, B01J 19/12	A1	(11) International Publication Number: WO 00/48731 (43) International Publication Date: 24 August 2000 (24.08.2000)
(21) International Application Number: PCT/GB00/00524 (22) International Filing Date: 17 February 2000 (17.02.2000) (30) Priority Data: 9903474.6 17 February 1999 (17.02.1999) GB (60) Parent Application or Grant UNIVERSITY OF NEWCASTLE [/]; O. RAMSHAW, Colin [/]; O. JACHUCK, Roshan, Jeet, Jee [/]; O. RAMSHAW, Colin [/]; O. JACHUCK, Roshan, Jeet, Jee [/]; O. HARRISON GODDARD FOOTE; O.	Published	
(54) Title: PROCESS FOR THE CONVERSION OF A FLUID PHASE SUBSTRATE BY DYNAMIC HETEROGENEOUS CONTACT WITH A SOLID AGENT (54) Titre: PROCEDE DE TRANSFORMATION D'UN SUBSTRAT EN PHASE FLUIDE PAR MISE EN CONTACT HETEROGENE DYNAMIQUE AVEC UN AGENT SOLIDE		
(57) Abstract <p>A process for the conversion of a substantially fluid phase substrate by heterogeneous contact of the substrate or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a surface of a support element or part thereof and the support element is adapted to rotate around an axis such that the solid phase agent provides a rotating surface or part thereof and the substrate provides a film flowing substantially radially outward from the axis in dynamic contact with the agent.</p> (57) Abrégé <p>L'invention concerne un procédé de transformation d'un substrat en phase sensiblement fluide par la mise en contact hétérogène du substrat ou d'un fragment ou d'un dérivé de celui-ci avec un agent en phase sensiblement solide, l'agent en phase solide étant une surface d'un élément de support ou d'une partie d'élément de support, et l'élément de support étant conçu pour tourner autour d'un axe de sorte que l'agent en phase solide constitue une surface tournante ou une partie de surface tournante, et le substrat constitue un film qui s'écoule sensiblement radialement en s'éloignant de l'axe et est en contact dynamique avec l'agent.</p>		

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(54) Title: PROCESS FOR THE CONVERSION OF A FLUID PHASE SUBSTRATE BY DYNAMIC HETEROGENEOUS CONTACT WITH A SOLID AGENT			
			
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Description

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PROCESS FOR THE CONVERSION OF A FLUID PHASE SUBSTRATE BY DYNAMIC HETEROGENEOUS CONTACT
WITH A SOLID AGENT

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5 The present invention relates to a process for the conversion of a substantially fluid
phase substrate by heterogeneous contact with a substantially solid phase agent, the
agent for use in the process, an apparatus therefor comprising the agent, novel agent
compositions, substrate compositions and the products thereof.

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10 In particular the present invention relates to a process for the conversion of a
substrate using an agent which is comprised as a rotating surface, the apparatus,
agent, and compositions for use therein and the products thereof.

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The invention makes use of rotating surfaces of revolution technology (hereinafter
RSORT) (commonly known as spinning disc technology).

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20 The spinning disc concept is an attempt to apply process intensification methods
within the fields of heat and mass transfer. The technology operates by the use of
high gravity fields created by rotation of a disc surface causing fluid introduced to the
disc surface at its axis to flow radially outward under the influence of centrifugal
acceleration in the form of thin often wavy films. Such thin films have been shown
to significantly improve the heat and mass transfer rates and mixing. The technology
was developed for typical heat and mass transfer operations such as heat exchanging,
heating, cooling and mixing, blending and the like, for example as disclosed in R J J
Jachuck and C Ramshaw, "Process Intensification: Heat transfer characteristics of
tailored rotating surfaces", Heat Recovery Systems & CHP, Vol. 14, No 5, p475-491,
1994.

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30 More recently the technology has been adapted for use as a reacting surface for
systems which are heat and mass transfer limited, for example for the reaction of
substrates which are highly viscous during at least a stage of the reaction and cause
problems in achieving good mixing and product yields.

45

35 Boodhoo, Jachuck & Ramshaw disclose in "Process Intensification: Spinning Disc
Polymeriser for the Manufacture of Polystyrene" the use of a spinning disc apparatus
in which monomer and initiator is reacted by conventional means to provide a pre-
polymer which is then passed across the surface of a spinning disc at elevated
temperature providing a conversion product in the form of polymerised styrene.

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EP 0 499 363 (Tioxide Group Services Limited) discloses another use for spinning disc technology in photo catalytic degradation of organic materials such a hydrocarbons. A solution of salicylic acid and titanium dioxide catalyst was passed
5 across the surface of a rotating disc and irradiated with ultra violet light.

15

These publications therefore disclose the use of spinning disc technology for heating and mass transfer in inert and reactive systems.

20

10 We have now surprisingly found that spinning disc technology may be further adapted to apply process intensification methods not only within the fields of heat and mass transfer but also within the field of heterogeneous contacting.

25

We have further surprisingly found that the quality of the product obtained is of higher quality than that obtained by conventional processing having, for example, a higher purity or, in polymers, a narrower molecular distribution.

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In addition to this, spinning disc technology can be used to obtain products not readily obtainable by other technology.

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20 Accordingly in its broadest aspect there is provided according to the present invention a process for the conversion of a substantially fluid phase substrate by heterogeneous contact of the substrate or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a surface of a support element or part thereof and the support element is adapted to rotate around an axis such that the solid phase agent provides a rotating surface or part thereof and the substrate provides a film flowing substantially radially outward from the axis in dynamic contact with the agent.

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30 Reference herein to a rotating surface is to any continuous or discrete planar or three dimensional surface or assembly which rotates approximately or truly about an axis, and preferably is reference to an approximate or true rotating surface of revolution. An approximate rotating surface of revolution may comprise an asymmetric axis and/or deviation in the surface body and/or circumference creating an axially or
35 radially undulating surface of revolution. A discrete surface may be in the form of a mesh, grid, corrugated surface and the like.

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5 Reference herein to a substantially radially outward flowing film as hereinbefore defined is to any fluid film which may be created by dynamic contact of the fluid
10 phase substrate and the rotating surface as hereinbefore defined, suitably the fluid phase substrate is contacted with the rotating surface at any one or more surface
5 locations and caused to flow outwardly by the action of centrifugal force. A film may be a continuous annulus or may be a non-continuous arc at any radial location. The substrate may provide a plurality of films in dynamic contact with a rotating
15 surface as hereinbefore defined.

10 Reference herein to an agent is to any agent which enables, facilitates or otherwise influences conversion of the fluid phase substrate by contact, and includes a
20 nucleation or growth agent adapted for fluid substrate conversion by phase change to form crystals or grow seed crystals, a reagent, catalyst or initiator adapted for fluid phase substrate conversion by reaction to form products, and the like.

15 The agent may be capable of being immobilised on the surface of a support element which is adapted to rotate as hereinbefore defined or may be comprised as an integral part of the surface as part of the construction thereof. The agent may be adapted to
25 achieve conversion by means of its composition or of its configuration, ie may be a chemical, electronic or like entity or may be a feature of shape.
20

Reference herein to a substrate is to the substrate to be converted or its fragment or derivative formed as part of the contacting with additional substrates, the rotating
30 surface or the agent.

35 The process of the present invention may be used for any desired heterogeneous conversion of one or more substrates, or of one or more fragments or derivatives thereof and is particularly useful for conversions which are heat and/or mass transfer
40 limited. By this means the process of the present invention is able to substantially improve process performance in terms of yield and selection of desired products by ensuring that heterogeneous contact is optimal and occurs at optimum conditions of
30 temperature, concentration, contact time and the like.

45 Preferably the process of the invention is a process for conversion of organics (e.g. hydrocarbons and other organic materials), such as prepolymerisation, polymerisation, copolymerisation, block polymerisation and the like, upgrading of
35 base hydrocarbons to provide petrochemicals for chemical, fuel or additives applications, for example etherification, dimerisation, alkylation, upgrading low
50

5 value higher chain (C_{10+}) to high value lower chain branched (C_{2-8}) hydrocarbons,
upgrading by decomposition (for example breakdown of salicylic acid) and the like.
The process is also admirably suited for adaptation of existing homogeneous
10 processes such as alkylation to form branched hydrocarbons and ethers, to
5 heterogeneous alternatives, using solid acids such as zeolites and the like.

Alternatively the process is a process for the crystallisation of a salt from a solution
15 formed prior to or during contact with the rotating surface.

10 The process may therefore comprise a conversion of one substrate with itself in the
presence of an agent as hereinbefore defined or may comprise the conversion of one
20 substrate with one or more additional substrates in the presence of an agent as
hereinbefore defined. Substrate(s) is fluid and may be gaseous, vaporous, liquid or
fine flowable solid.

15 The conversion may be carried out entirely or in part under the influence of an added
energy source which may be a source of thermal or radiation energy such as ultra
violet, micro wave, infra-red, x-ray, ultrasound or at elevated pressure or under the
25 influence of electric or magnetic fields.

20 The process of the invention as hereinbefore defined may operated in a single or
plural stages. A plural stage conversion may comprise a first pre-conversion stage
with further post conversion or upgrading stages, and may be carried out batchwise
35 with use of a single rotating surface as hereinbefore defined or may be carried out in
25 continuous manner with multiple rotating surfaces in series.

The process is particularly suited to continuous conversion, and may provide means
40 to perform hitherto batch wise conversions in continuous manner, by means of the
improved conversion efficiency and regulated contact time obtained with the
30 invention.

45 For example conversions requiring extended contact time may be carried out in
continuous manner with use of a recycle of fluid exiting at the periphery of the
rotating disc towards the axis of the rotating disc enabling sequential passes of fluid
35 across the surface. In continuous steady state operation an amount of fluid exiting
the disc may be drawn off as product and an amount may be returned by recycle for
50 further conversion with an amount of fresh substrate feed.

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Second or more substrates may be added to the substrate as it passes from one rotating assembly to the next or be added directly to the rotating assembly anywhere between the axis of rotation or the exit from the assembly. In certain cases a multi-step conversion process may be achieved by substrate addition or additions between the axis of rotation and the exit of a single rotating assembly to achieve more than one conversion step in a single pass. It is also possible to have more than one agent on the surface either intermixed or at different radial locations in order to facilitate more than one conversion or conversion stage. These different sections may be controlled at different temperatures and conditions and have different surface geometries as appropriate to the process needs.

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Conversion may be carried out with initiation and/or quench a known in the art. It is of a particular advantage of the invention that a plural stage process may employ a pre-mixing stage enabling rapid blending of any diluents or solvent with substrate, or of plural substrates, followed by a conversion stage as hereinbefore defined and a post-conversion quench stage in which reacted product exits the rotating surface at the periphery thereof and is collected in a suitable collection means or on a third stage rotating surface on which quench may be performed by the blending of a quench medium or by cooling. The process of the invention enables rapid homogeneous quenching.

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It will be apparent that the process of the invention may be controlled both by selection of a specific rotating surface for use as agent support, by nature of immobilisation of agent thereon and by selecting process variables such as temperature, speed of rotation, rate of substrate feed, conversion time and the like. Accordingly the process of the invention provides enhanced flexibility in process control including both conventional control by means of operating conditions, and additionally control by means of rotating surface type and nature of agent immobilisation.

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Agents may be immobilised on the rotating surface by any means known in the art for supporting agents, suitably by means of spraying such as plasma spraying of solutions of metals, sol gel impregnating with drying and optional calcining or reduction and the like. Agent may be mechanically or chemically immobilised or both. Preferably agent is mechanically coated and chemically immobilised to interact

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5 with and bond with the rotating surface in manner to ensure coherent coating with any desired distribution and loading of agent.

10 Agents may be supported directly on the rotating surface or may be supported on a
5 surface coating of a material adapted to provide surface features or enhanced surface area. For example the rotating surface may be provided with enhanced surface area in the form of macro surface features such a corrugated continuous surface for
15 example of a metal foil and the like or micro features such as a coating of microporous material such as zeolite or refractory oxide and the like. The agent may
10 be provided with any additional coating to enhance its conversion performance.

20 The method of preparing the agent may also be selected according to the nature of the agent itself.

15 Accordingly coating the rotating surface with agent by spraying is particular suited in
25 case of agents in the form of metal catalyst such as metal or alloy powder which are commonly used for example in hydrogenation reactions and the like.

30 Coating with a sol gel is particularly suited for use with agents in the form of
20 materials which readily form sol gels, such as inorganic oxide catalysts which are commonly used for example in oxidation and partial oxidation reactions.

35 The coating techniques may be used as commonly known in the art, with use of known spray apparatus and dipping methods. In the case of non planar rotating
25 surfaces or rotating surfaces with additional surface features it may be desired to adapt the known techniques to ensure uniformity of coating, and avoid any distortion by virtue of gravitational, meniscal, capillary effects and the like. These effects may
40 suitably be overcome with known methods in which an electrostatic field is applied to one object to be coated during the coating process, or with sol gel techniques in
30 which the colloidal structure of the solution comprising a suspension of solid particles of colloidal dimensions in a liquid is less prone to gravitational effects, meniscal or capillary effects, optionally additionally mounting the rotating surface in
45 manner to allow vertical rotation during drying and calcining.

35 A RSORT apparatus (commonly known as a spinning disc reactor) generally includes
50 within a conversion chamber a rotating surface or an assembly of a plurality of these

5 which is rotated about an axis to effect transfer of one or more substrates from the axis preferably radially across the rotating surface.

10 A RSORT apparatus as hereinbefore defined comprising a rotating surface as
5 hereinbefore defined has a number of advantageous constructional features according to the present invention.

15 Any suitable feed means may be provided to feed the at least one substrate onto the rotating surface. For example, the feed means may comprise a feed distributor in the
10 form of a "shower head", a "necklace" of outlets for a simple, preferably adjustable, single point introduction such as a "hose-pipe type" feed means. Preferably, the feed
20 means comprises a feed distributor having a plurality of uniformly spaced outlets for the at least one substrate on to the rotating surface as hereinbefore defined.

15 The feed means may be provided at any suitable position with respect to the rotating surface which allows feed of the substrate. For example, the feed means may be
25 axially aligned with the rotating surface for axial feed. Alternatively, the feed means may be positioned such that the feed is spaced from the axis of the rotating surface. Such a position may lead to more turbulence and an enhanced mixing effect. In a
30 further alternative embodiment, feed means may comprise a single feed to a trough which is preferably situated on or co-axial with the axis of rotation of the rotating surface. In this alternative embodiment, substrate flows from the feed outlet into the
35 trough and is subsequently spread out of the trough on to the rotating surface by centripetal force. In a preferred embodiment, the rotating element as hereinbefore
25 defined comprises a trough situated on the axis of rotation.

40 The trough as hereinbefore defined may be of any suitable shape such as continuous or annular. For example it may have a continuous concave surface comprising part of a sphere, such as a hemispherical surface or it may have an inner surface joined to
30 the rotating surface by at least one connection wall or at least two, in the case where the trough is annular. The inner surface and connection wall may be of any form which allows the function of a trough to be fulfilled. For example the inner surface
45 may be parallel to the rotating surface or concave or convex. The connection wall may comprise a single circular or ovoid wall or a plurality of straight walls. The
35 walls may diverge or converge towards the rotating surface.

5 Preferably, a single circular wall is provided which converges towards the rotating surface to form an undercut trough. This shape generates a reservoir which enhances a circumferential distribution of the substrate flow.

10 The trough may be lined, coated or otherwise provided with a solid phase agent as hereinbefore described, such as a catalytic material.

15 Advantageously, a matrix may be provided in the trough so as to help reactant present in the trough to rotate with the support element, thereby helping to achieve substantially uniform flow from the trough across the first surface. The matrix may be in the form of a plug of fibrous mesh, such as metal or plastics wool, or may take 20 the form of a plurality of projections which are secured to an inner surface of the trough. Other matrix means will be apparent to the skilled reader. In some embodiments, the matrix is manufactured of a material which is inert with respect to the at least one reactant or the product and which is not significantly affected by temperature and other variable process conditions. Alternatively, the matrix may be 25 made of or coated with a material which does interact with the at least one reactant or the product, such as a heterogeneous catalyst (e.g. nickel, palladium or platinum or any suitable metal or alloy or compound thereof) or any of the solid phase agents as hereinbefore described. Where the matrix is made out of an electrically conductive material, it may be possible to supply an electric current therethrough and thus to 30 provide heating means for heating the at least one reactant within the trough.

35 Any suitable collection means may be provided for collection of the product as it leaves the rotating surface at its periphery. For example, a receptacle in the form of a bowl or trough at least partially surrounding the rotating element or other fixed part 25 of the apparatus. The collection means may additionally comprise a deflector positioned around the periphery of the rotating surface to deflect product into the collection means. The deflector is preferably positioned at an acute angle to the 40 rotating surface.

30 The components of the collection means, such as the bowl or trough or deflector, 45 may be coated or otherwise provided with a solid phase agent as hereinbefore described, such as a heterogeneous catalyst appropriate to the fluid phase substrate being processed on the support element, or may even consist entirely of a material which acts as a heterogeneous catalyst. Furthermore, the components of the 50 collection means may be heated or cooled to a predetermined temperature so as to 35

5 enable control over reaction parameters, for example by serving to halt the processing
of substrate as this leaves the surface of the support element. Feed means for
supplying a further substrate to the substrate leaving the surface may also be
10 provided. For example, there may be provided feed means for feeding a quenching
5 medium to substrate in the collection means so as to halt chemical or other reactions
of the substrate when this has left the surface.

15 The collection means may further comprise outlet means of any suitable form. For
example, there may be a single collection trough running around the periphery of the
10 disc or a collection bowl partially surrounding the rotating element.

20 Outlet means may also be provided in the collection means and these may take the
form of apertures of any size and form situated at any suitable position of the
collection means to allow egress of the product. In one preferred embodiment, the
15 outlet means are situated to allow vertical egress of the substrate in use.

25 The apparatus may further comprise any suitable control system. Such a control
system may regulate the temperature or contact time of substrate and agent by means
of speed of rotation, rate of substrate feed and other process parameters to obtain an
20 optimum result.

30 The apparatus as hereinbefore defined may comprise means for optimising
conversion conditions. For example, means for imparting an additional movement to
the rotating surface, and thus to the substrate, may be provided. Such movement
35 could be in any desired plane or plurality of planes and preferably comprises
25 vibration. Any suitable vibration means may be provided, such as flexible mounting
of the surface or off centre mounting, both inducing passive vibration or active
vibration means, such as a mechanical element in contact with the rotating element
40 and vibrating in a direction parallel to the rotating element axis. Preferably a passive
30 vibration means is provided in the form of off centre mounting of the rotating
element on its axis of rotation. Vibration may alternatively be provided by an
ultrasonic emitter in contact with the rotating element for vibration in any desired
45 plane or plurality of planes.

50 35 The rotating surface may have any shape and surface formation to optimise
conversion conditions. For example the rotating surface may be generally planar or

5 curved, frilled, corrugated or bent. The rotating surface may form a cone or be of generally frusto conical shape.

10 In one preferred embodiment the rotating surface is generally planar and preferably generally circular. The periphery of the rotating surface may form an oval, rectangle or other shape.

15 In another preferred embodiment the rotating surface is provided as the inner surface of a cone. The apparatus may comprise at least one cone and at least one other rotating surface or at least one pair of facing cones positioned so as to allow a two stage process of pre-conversion and conversion or pre-blending and conversion with one or more substrates fed to each cone. Preferably substrates or pre-reacted substrate exits a smaller cone (or other surface of rotation) in a spray on to the surface of a larger cone (or other surface of rotation) by which it is at least partially surrounded and for the surface of which a further substrate is fed by feed means as hereinbefore defined, to allow mix of the two substrates on the larger rotating surface. Preferably, means are provided such that the two cones counter rotate. Such an arrangement enhances mixing and intimate contact of the substrates and reduces the required physical contact time. Alternatively, means are provided such that the cones co-rotate or one is stationary.

30 A rotating surface of any shape and surface formation as hereinbefore defined may be provided with surface features which, in addition to the immobilised agent, serve to promote conversion. For example, the surface may be micro or macro profiled, micro or macro porous, non stick, for example may have a release coating, may be continuous or discontinuous and may comprise elements such as mesh, for example woven mesh, reticulate foam, pellets, cloth, pins or wires, for enhanced surface area, enhanced or reduced friction effect, enhanced or reduced laminar flow, shear mixing of recirculation flow in axial direction and the like.

35 In one preferred embodiment, mixing characteristics of the rotating surface are enhanced by the above features or the like provided on or in the rotating surface. These may be provided in any suitable regular or random arrangement of grids, concentric rings, spider web or like patterns which may be suitable for a given application.

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Alternatively or additionally to any other surface feature, radially spaced pins in the form of circles or segments of circles may be provided.

10

5 In another preferred embodiment, a porous surface coating is provided, which aids conversions of certain substrates. Such a coating may be provided in combination with any other of the aforementioned surface features.

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10 Surface features in the form of grooves may be concentric or may be of any desired radially spaced form. For example, the grooves may form "wavy" or distorted circles for maximised mixing.

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Grooves may be parallel sided, or may have one or both sides which diverge to form undercut grooves or which converge to form tapered grooves. Preferably, the grooves are undercut to promote mixing.

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Grooves may be angled to project towards or away from the axis of the rotating surface to enhance or reduce undercut or taper.

30

20 Energy transfer means may be provided for the rotating surface or substrate as hereinbefore described. For example heating means may be provided to heat the substrate, for example, as part of the feed means. Additionally, or alternatively heating means may be provided to heat the rotating element in the form of radiant or other heaters positioned on the face of the rotating element which does not comprise the rotating surface for conversion. Preferably, radially spaced, generally circular
25 radiant heaters are provided.

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Any preferred cooling or quenching means may be provided in a suitable position to cool the reacted substrate. For example cooling coils or a heat sink may provide cooling by heat exchange, or a reservoir of quench may provide cooling or reaction
30 termination by intimate mixing in the collection means.

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Cooling means for the substrate may be provided in any suitable position, such as on, in or near the deflector or collection means.

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35 Multiple rotating elements may be provided in an apparatus as hereinbefore defined. For example the rotating elements may be provided in series within the apparatus, with reacted substrate from the first disc being fed directly to at least one further disc.

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Alternative or additionally, rotating elements may be provided in parallel with a single reservoir of substrate fed simultaneously to a plurality of discs. In this preferred embodiment a single collection means may be envisaged. The invention is now illustrated in non-limiting manner with reference to the following figures and examples.

Example 1 – Polymerisation of ethylene using a catalyst coated disc

Phillips catalyst was coated onto the surface of a spinning disc apparatus using methods as described hereinbefore. The coated disc was mounted in a spinning disc apparatus.

The spinning disc apparatus used is shown in diagrammatic form in Figure 1. The main components of interest being:

- i) Top Disc – A smooth brass disc of thickness 17mm and diameter 500mm capable of rotating around a vertical axis.
- ii) Liquid Distributor – A circular copper pipe of diameter 100mm, positioned concentrically over the disc, sprayed fluid vertically onto the disc surface from 50 uniformly spaced holes in the underside. Flowrate was controlled manually by a valve and monitored using a metric 18 size, stainless steel float rotameter. A typical fluid flow rate was 31.3cc/s.
- iii) Motor – A variable speed d.c. motor capable of rotating at 3000 rpm was used. The rotational speed was varied using a digital controller calibrated for disc speeds between 0 and 1 000 rpm. A typical rotational speed was 50rpm.
- iv) Radiant Heaters – 3 radiant heaters (each consisting of two elements) spaced equally below the disc provided heat to the disc. The temperature was varied using a temperature controller for each heater. Each heater temperature could be controlled up to 400°C. Triac regulators were used to control the speed of the controller response. (These remained on setting 10 throughout the tests).
- v) Thermocouples and Datascaner – 16 K-type thermocouples embedded in the top disc gave an indication of the surface temperature profile along the disc radius. Odd numbered thermocouples 1 to 15 inclusive were embedded from underneath the disc to a distance 3mm from the upper disc surface. Even numbered thermocouples, 2 to 16 inclusive were embedded from underneath the disc to a distance 10mm from

the upper disc surface. Each pair of thermocouple, ie 1 & 2 and 3 & 4 and 5 & 6 etc, were embedded adjacently at radial distances of 85mm, 95mm, 110mm, 128mm, 150mm, 175mm, 205mm and 245mm respectively (see Figure 3). The thermocouples were connected to the datascanner which transmitted and logged the data to the PC at set intervals using the DALITE Configuration and Monitoring Software Package.

vi) Manual Thermocouple – A hand-held K-type thermocouple was used to measure the bulk fluid temperature on top of the disc.

The rig was used in two arrangements. In one arrangement, feed was constantly added and the heated product was sent to the collection trough. In an alternative arrangement the rig was assembled with a recycle. The fluid flowpath in the recycle arrangement is shown in Figure 4.

The spinning disc apparatus of Figure 1 was started up and temperature and rotational speed adjusted. When steady stage was achieved gaseous ethylene was fed to the revolving catalyst coated disc surface at it axis. Product was withdrawn in the collection trough at the periphery of the disc. Analysis revealed the product was high grade polyethylene.

Example 2 – Precipitation of barium sulphate crystals using a disc providing a nucleation/growth rotating surface

A nucleation surface was provided on the surface of a disc adapted to provide a rotating surface using methods as described hereinbefore. The disc was mounted in a spinning disc apparatus.

The spinning disc apparatus used is as described in Example 1. Barium chloride and sodium sulphate solutions were reacted on the surface of the spinning disc. Barium sulphate crystals precipitated out from the solution and the effect of rotational speed on the crystal size and size distribution was studied.

Example 3 – Precipitation of calcium carbonate crystals using a disc providing a nucleation/growth rotating surface

A nucleation surface was provided on the surface of a disc adapted to provide a rotating surface using methods as described hereinbefore. The disc was mounted in a spinning disc apparatus.

5 The spinning disc apparatus used is as described in Example 1. Calcium hydroxide and carbon dioxide (gas/liquid) were reacted on the surface of the disc and the effect of rotational speed on the crystal size and size distribution was studied.

10 5 Figure 1 illustrates a conventional spinning disc apparatus which is suited for adaptation according to the present invention. The apparatus is enclosed in vessel (1) having at its axis a drive shaft (2) supporting a spinning disc (3). Feed means (4) provides substrate to the surface of the disc (5) at its axis (6). Rotation of the disc causes substrate to flow radially outwards, whereby it contacts the surface of the spinning disc. Fluid is collected at the peripheral edges of the disc by means of collection trough (7) and may be rapidly quenched by means of cooling coils (8). A skirt (9) prevents meniscal draw back of fluid contaminating the drive shaft mechanism. Inlet means (10) enable controlled environment conditions to be provided, for example a nitrogen atmosphere. Outlet vent means (11) enable the venting of atmospheric gases or gases evolved during operation. Observation means are provided by means of windows (12) to observe the progress of the conversion.

25 In Figure 2 is shown a typical spinning disc in the form of a rotating surface according to the present invention comprising an immobilised agent on its surface.

30 20 The disc (3) is illustrated without any additional surface features and comprises uniform coating (31) which is fused by chemical interaction (32) with the disc surface (33).

35 In Figure 3 is shown an alternative spinning disc in which the agent which it is desired to immobilise is also known as a conventional support material, for example a refractory oxide. In this case the spinning disc (3) is constructed entirely of refractory oxide, the body of which (34) provides a support function and the surface of which (35) provides a catalytic function.

40 30 In the process of the invention an apparatus of Figure 1 is started up and operated as described in Example 1. In the case that the process is an exothermic conversion, cooling coils (8) may be used to quench the collected product in the trough (7). The spinning disc is provided with heating coils (not shown) which may be used to initiate or maintain conversion. The disc or the reactor vessel may be provided with a source of radiation, means for applying an electric or magnetic field and the like as described, at or above the disc surface or at the wall of the reactor vessel.

5 Figure 4 shows an alternative reactor including a spinning disc (3) mounted on a
drive shaft (2) connected to a motor (40). Feed means (4) supplies reactant to a
10 central portion of the surface (5) of the disc (3), and collector means (7) collects
product thrown from the surface (5). The product is taken to a feed tank (41) from
5 where it is pumped by pump (42), through a flowrate control valve (43) and
rotameter (44) before being recycled to the feed (4). Electric heaters (45) and
thermocouples (46) are provided underneath the disc (3) and connected to a
15 datascanner (47) and a control unit (48) which serves to regulate the temperature of
the disc (3) and its rotational speed. A datalogger (49) is provided to record process
10 details such as temperatures and rotational speeds.

20 In Figure 5 there is shown an axially located central trough (14) which is continuous
and forms a well situated on the axis of rotation (6) of the rotating surface (5) of a
disc (3). Rotation causes reactant (15) supplied by the feed means (4) to flow to the
15 wall and form an annular film (16) within the trough (14). The annular film (16) then
spills over onto the surface (5) of the disc (3) to form a film (17) on the surface (5).
25

In Figure 6 the trough (13) is annular and forms a channel co-axial about the axis of
rotation (6) of the disc (3). Rotation assisted by the trough profile causes reactant
30 (15) to flow into the trough (13) and to the wall thereof and form an annular film (16)
within the trough (13) before spilling over onto the surface (5) of the disc (3) in the
form of a film (17).
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35 Further advantages of the invention are apparent from the foregoing.

Claims

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CLAIMS:

1. A process for the conversion of a substantially fluid phase substrate by heterogeneous contact of the substrate or a fragment or derivative thereof with a substantially solid phase agent wherein the solid phase agent is comprised as a surface of a support element or part thereof and the support element is adapted to rotate around an axis such that the solid phase agent provides a rotating surface or part thereof and the substrate provides a film flowing substantially radially outward from the axis in dynamic contact with the agent.

2. A process according to claim 1, wherein the solid phase agent is in the form of a mesh, grid or corrugated surface.

3. A process according to claim 1 or 2, wherein the solid phase agent comprises a nucleation or growth agent adapted for fluid phase substrate conversion by phase change to form crystals or grow seed crystals.

4. A process according to claim 1 or 2, wherein the solid phase agent comprises a reagent, catalyst or initiator adapted for fluid phase substrate conversion by reaction to form products.

5. A process according to claim 4, wherein the solid phase agent is a zeolite.

6. A process according to claim 4, wherein the solid phase agent is a refractory oxide.

7. A process according to claim 4, wherein the solid phase agent is a sol gel.

8. A process according to claim 4, wherein the solid phase agent is Phillips catalyst.

9. A process according to any preceding claim, wherein additional thermal or radiation energy, including UV, IR, microwave, RF, X-ray, ultrasound, electric fields and magnetic fields, is applied to the substrate.

10. A process according to claim 9, wherein the additional energy is applied to the substrate when on the rotating surface.

5

11. A process according to claim 9 or 10, wherein the additional energy is applied to the substrate as it is being supplied to the rotating surface.

10

5 12. A process according to claim 9, 10 or 11, wherein the additional energy is applied to the substrate after it has flowed across the rotating surface.

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13. A process according to any preceding claim, wherein the rotating surface is mechanically vibrated.

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14. A process according to claim 13, wherein the rotating surface is mounted off-centre on the axis of rotation.

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15. A process according to claim 13 or 14, wherein the surface is flexibly mounted on the support element.

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16. A process according to claim 13, 14 or 15, wherein a mechanical vibrator is attached to the surface or the support element.

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20 17. A process according to any one of claims 3 to 16, wherein the solid phase agent is sprayed onto the surface.

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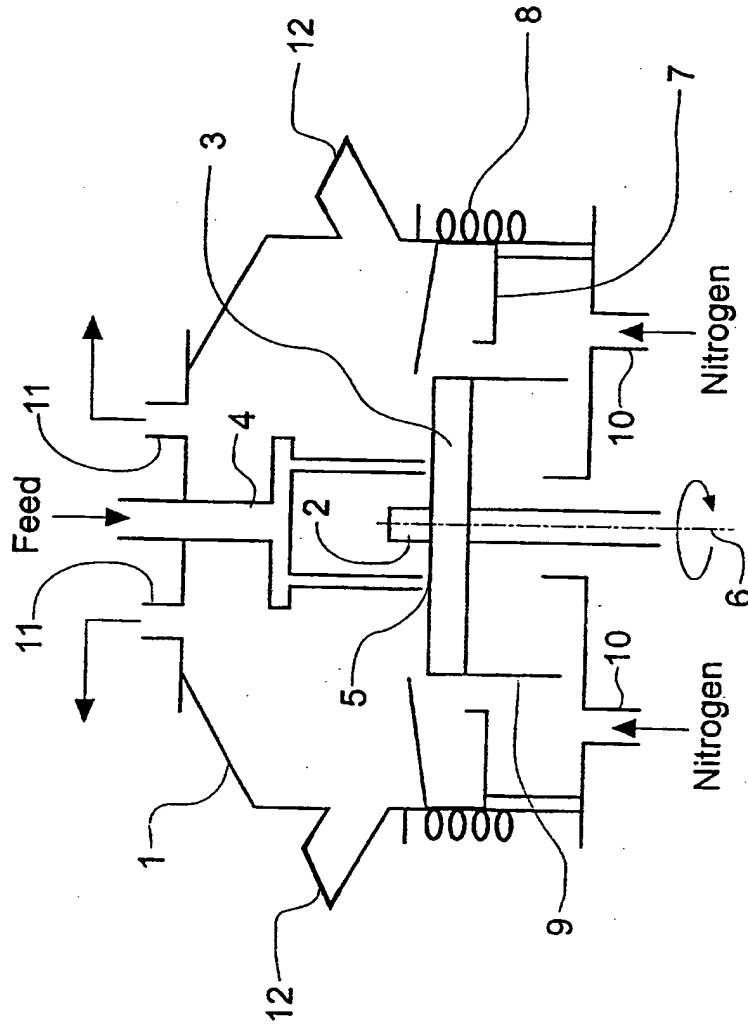


Fig. 1

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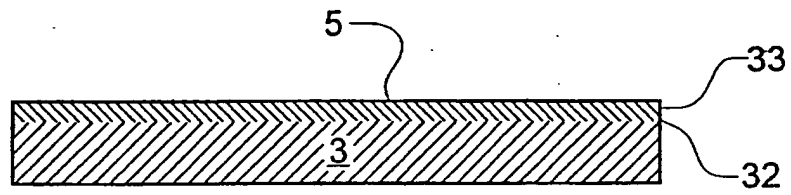


Fig. 2

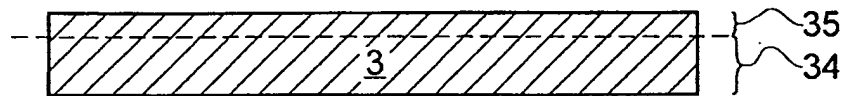


Fig. 3

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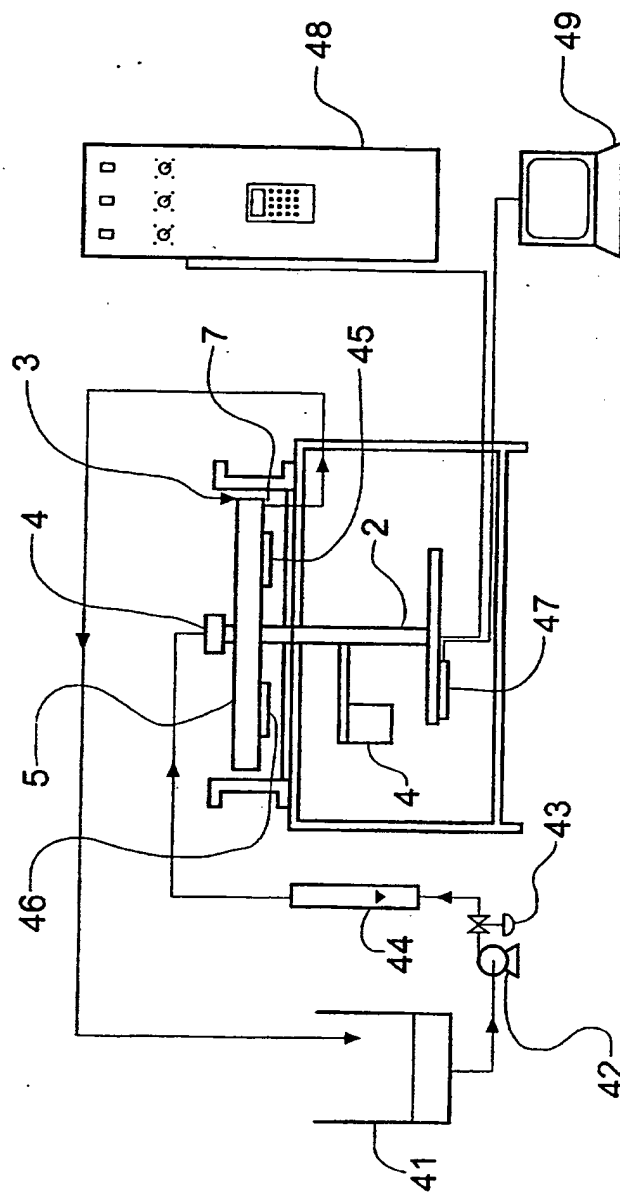


Fig. 4

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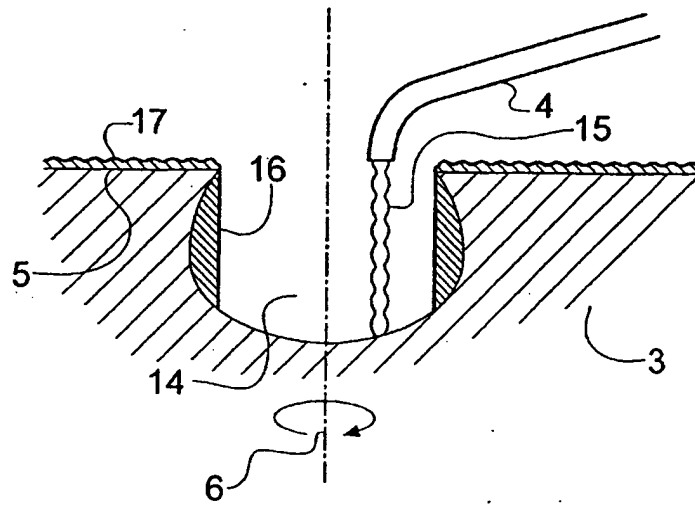


Fig. 5

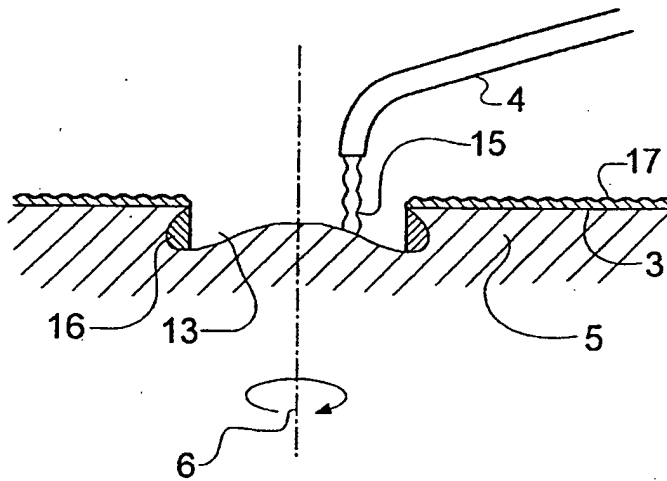


Fig. 6

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/GB 00/00524

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01J19/18 B01J19/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO 96 00189 A (CHRISTENSEN PAUL A, HUTSON GRAHAM V) 4 January 1996 (1996-01-04) page 11, line 3 -page 14, line 5 page 19, line 25 -page 21, line 7 claims 1,13-15,21,22,27-33; figures 3,4	1,3,4,6, 7,9-11
A	US 4 356 133 A (COWEN GEOFFREY, NORTON-BERRY PHILIP, STEEL MARGARET L) 26 October 1982 (1982-10-26) column 2, line 1 - line 6 column 3, line 53 -column 4, line 13 column 4, line 53 -column 5, line 11 figure 1	1,4,9-12
-/-		



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"Z" document member of the same patent family

Date of the actual completion of the international search

22 June 2000

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/00524

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